

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. :10/595,686  
Applicants :Jin Ho CHOY et al.  
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Examiner :Rebecca Y. Lee  
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**DECLARATION UNDER 37 CFR §1.132**

Commissioner for Patents  
P. O. Box 1450  
Alexandria, VA 22313-1450

Dear Sir:

I, Jin-Ho Choy, declare that:

1. I am a distinguished professor at Ewha Womans University of Korea. I graduated from Yonsei University of Korea and got Ph.D. in chemistry from Munich University.
  
2. I have been working as An Int'l Editorial Board Member of "International Journal of Inorganic Materials" (Elsevier Sciences), which is now merged to the "Solid State Sciences" since 1998. Also I have been working as advisory board member for "Journal of Solid State Chemistry"(Elsevier) , " Chemistry of Materials" (American Chemical Society, USA) and " Multidiscipline Modelling in Materials and Structure(MMMS)" (VSP-Brill,Netherland).

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3. I am familiar with the subject matter described and claimed in the United States Patent Application Serial No. 10/595,686, filed on December 27, 2006, entitled "Method for Forming ZnO Nano-Array and ZnO Nanowall for UV Laser on Silicon Substrate" ("Present Application")

4. I have read and am familiar with the Office Action issued on February 1, 2010 including the scientific literatures cited therein: Tian et al., Biometric Arrays of Oriented helical ZnO nanorods and Columns, JACS 2002, 124, 12954-12955 ("Tian"); Boyle et al., "Novel low temperature solution deposition of perpendicularly oriented rods of ZnO: substrate effects and evidence of the importance counter-ions in the control of crystal growth, Chem. Commun, 2002, 80-81 ("Boyle"); and U.S. Patent No. 7,294,417 issued to Ren et al. ("Ren"). It is my understanding that claims 1, 4, 6 and 10 are rejected under 35 USC 103(a) as unpatentable over Tian in view of Boyle; claims 1, 4 and 6 are rejected under 35 USC 103(a) as unpatentable over Boyle; claim 10 is rejected under 35 USC 103(a) as unpatentable over Boyle in view of Tian; and claims 2, 5, 7 and 13 are rejected under 35 USC 103(a) as unpatentable over Tian in view of Boyle.

5. I understand that claims 1 and 2, among the rejected claims, are independent claims. Claim 1 is directed to a method of forming a ZnO nanorod array, which comprises coating on a substrate ZnO nanoparticles serving both as a buffer layer and a seed layer; and growing the ZnO nanoparticles into crystals to form the ZnO nanorod array in a nutrient solution consisting of hexamethylenetetramine, at least one selected from the group consisting of Zn nitrate, Zn acetate, and a derivative thereof, and a solvent.

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wherein the substrate is made of Si, and wherein the operation of growing the ZnO nanoparticles in the nutrient solution is performed at 90 to 100 °C.

Claim 2 is drawn to a method of forming a ZnO nanowall array, which comprises coating on a substrate ZnO nanoparticles serving both as a buffer layer and a seed layer; and growing the ZnO nanoparticles into crystals to form the ZnO nanowall array in a nutrient solution consisting of sodium citrate, at least one selected from the group consisting of Zn acetate and its derivative, and a solvent,

wherein the substrate is made of Si, sapphire ( $\text{Al}_2\text{O}_3$ ), GaN,  $\text{ScAlMgO}_4$ , or  $\text{LiNbO}_3$ , and wherein the operation of growing the ZnO nanoparticles in the nutrient solution is performed at 90 to 100°C.

6. It is my understanding that Tian discloses synthesis of unusually extended and oriented helical nanostructures in synthetic ceramics. See page 12954, left column, lines 7-8. In more particularly, Tian describes making oriented ZnO rods and using them as the base material to grow the helical structures. See Id., at lines 25-27. The nutrient solution used in Tian to grow the helical ZnO nanostructures contains hexamethylenetetramine ("HMT") (0.10M), sodium citrate (0.0010M) and Zn nitrate (0.030M).

7. I note that the nutrition solution to be used in the method of claim 1 differs from that disclosed in Tian in that it does not use sodium citrate. I also understand that the specification and the drawings of the Present Application demonstrate that the method of claim 1 using such nutrition solution can produce a perfectly oriented ZnO nanorod array. More particularly, Fig. 3 of the Present Application is a powder X-ray

diffraction pattern of a ZnO nanorod array formed on a Si-wafer using a nutrient solution containing 0.1M Zn nitrate solution and 0.1M HMT without containing sodium citrate.

Fig. 3 shows the [00l] peaks only without any of the [h00] peaks. In my opinion, Fig. 3 clearly evidences the ZnO nanorod produced has the perfect orientation. In comparison, Fig. 1C of Tian shows that the ZnO nanorod prepared by the method disclosed in Tian is imperfectly oriented and has a helical structure. I understand that the test demonstrated in Fig. 3 uses a Si-wafer as a substrate. Therefore, it is my opinion that producing perfectly oriented ZnO nanorods according to the method of claim 1 would have been unexpected given the disclosure of Tian that the nutrition solution containing HMT, Zn nitrate and sodium citrate only produced imperfectly oriented ZnO nanorods and lack of any guidance in Tian regarding how to produce perfectly oriented ZnO nanorods.

8. Boyle discloses a new approach for the low temperature solution growth of perpendicularly oriented ZnO rods on a TO-glass substrate. The procedure involves (1) formation of a CBD ZnO template layer of the desired morphology (obtained via judicious choice of ligand and counter-ion) and (2) subsequent growth of acicular rods on the templates. See page 80, left column, lines 4 from the bottom to right column, line 2. According to Boyle, "control of the density of the ZnO rod arrays is achieved through the first step while dimensionality and crystallinity are tailored via the second step." Id. Boyle further states that the rationale behind the second step derives from the need to impede the propensity of ZnO crystallites to undergo basal twinning and to enhance the crystallinity and growth of ZnO rods. See page 80, right column, lines 17-20. Boyle

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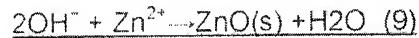
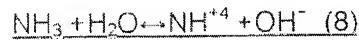
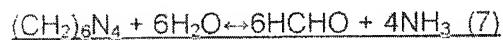
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finally observes that "the best results were obtained for heated aqueous solutions containing zinc acetate, HMT and additional chloride ion (the latter appears to impede twinning.)" See Id. at lines 20-23.

9. Thus, given the knowledge in the relevant area at the time the claim invention was made, it is my opinion that producing the ZnO nanorods or ZnO nanowalls with the perfect orientation according to the methods of claims 1 and 2 would have been unexpected to one skilled in the art in comparison with the teachings of Tian and/or Boyle.

10. I understand that the nutrition solution used in claim 2 does not contain HMT. I note that both Tian and Boyle use HMT in their nutrition solution. Related arts discloses that the role of HMT is to supply the hydroxyl ion to drive the precipitation reaction as shown below (Sci. Technol. Adv. Mater. 10 (2009) 013001, page 7).



In other words, HMT is critical or essential component to grow ZnO nanoparticle. Thus, it is my opinion that one of ordinary skill in the art at the time of invention would have understood that HMT is an essential component of a nutrition solution for growing ZnO nanoparticles. As a result, I opined that growing ZnO nanoparticles using the nutrition solution without containing HMT would have been unexpected to one skilled in the art.

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11. Furthermore, the nanowall arrays prepared by the method of claim 2 show UV laser threshold of 5kw/cm, which is about 90% lower than the lowest threshold obtained in currently available nanorod arrays.

This unexpected result is based on the extraordinary crystal structure in which the c-plane of ZnO nanowalls lies perpendicular to the substrate. With this perpendicular orientation, ZnO nanowalls can work as optical waveguide and also trapping agent. In contrast to this perpendicular orientation of the c-plane for ZnO nanowalls of the present invention, the nanowall structure developed by Ren as in US Patent 7294417, has the c-plane of ZnO crystal parallel with the substrate. The difference in the orientations of c-plane for ZnO nanowalls against the substrate can be easily identified from the XRD patterns in Fig. 17A of Ren(US Patent 7294417) and Fig. 9 of the present invention. Ren's XRD pattern in Fig. 17A shows both the diffraction peaks from (002) crystal plane for ZnO and (110) crystal ab-plane for sapphire and reveals the epitaxial relation between the c-plane of ZnO nanowalls and the ab-plane of the sapphire substrate as described by Ren in column 18, lines 65-67 to column 19, line 1 of US Patent 7294417.

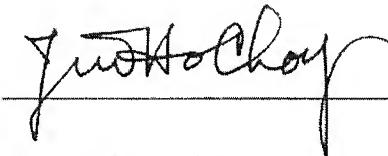
Although the external appearance looks similar as a nanowall, the crystal structures, which is essential part in optical applications, are totally different in the present invention and Ren's.

Therefore, it is not likely to conceive the method for crystal structure of the present invention from the Ren's results.

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12. The undersigned further declares that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signature



Name

Jin-Ho Choy

Date

May 24<sup>th</sup>, 2010